

# SEARCH REQUEST FORM

Requestor's  
Name:

Chaney

Serial

Number:

09/879 633

Date:

1/24/04

Phone:

~~2128~~  
21824

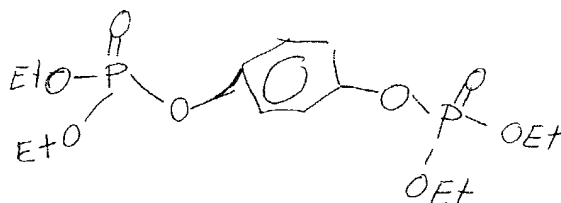
Art Unit:

1745

## Search Topic:

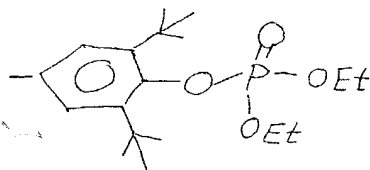
Please write a detailed statement of search topic. Describe specifically as possible the subject matter to be searched. Define any terms that may have a special meaning. Give examples or relevant citations, authors keywords, etc., if known. For sequences, please attach a copy of the sequence. You may include a copy of the broadest and/or most relevant claim(s).

Please search for the 2 compounds



benzene-1,4-bis-diethylphosphate

OR



diethyl (2,6-di-tert-butyl-4-methylphenyl) phosphate,  
-see attached- especially.

used as flame retardants in battery electrolytes

Chem Abstracts doesn't seem to use the names given <sup>here</sup> for the compounds.

## STAFF USE ONLY

Date completed:

1/28/04

Searcher:

Sheppard

Terminal time:

Elapsed time:

CPU time:

Total time:

Number of Searches:

Number of Databases:

### Search Site

\_\_\_\_\_ STIC

\_\_\_\_\_ CM-1

\_\_\_\_\_ Pre-S

### Type of Search

\_\_\_\_\_ N.A. Sequence

\_\_\_\_\_ A.A. Sequence

\_\_\_\_\_ Structure

\_\_\_\_\_ Bibliographic

### Vendors

\_\_\_\_\_ IG Suite

\_\_\_\_\_ STN

\_\_\_\_\_ Dialog

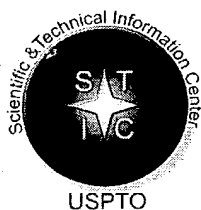
\_\_\_\_\_ APS

\_\_\_\_\_ Geninfo

\_\_\_\_\_ SDC

\_\_\_\_\_ DARC/Questel

\_\_\_\_\_ Other



# STIC Search Report

## Biotech-Chem Library

STIC Database Tracking Number: 112899

TO: Carol Chaney

Location:

Art Unit: 1745

January 28, 2004

*Rem  
6 C 81*

Case Serial Number: 09/879633

From: P. Sheppard

Location: Remsen Building

Phone: (571) 272-2529

sheppard@uspto.gov

### Search Notes

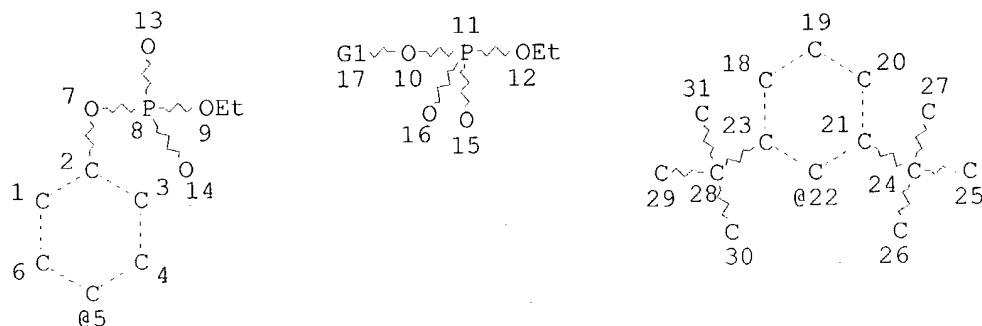
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 FILE 'HCAPLUS' ENTERED AT 12:29:04 ON 28 JAN 2004  
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 FILE LAST UPDATED: 27 Jan 2004 (20040127/ED)

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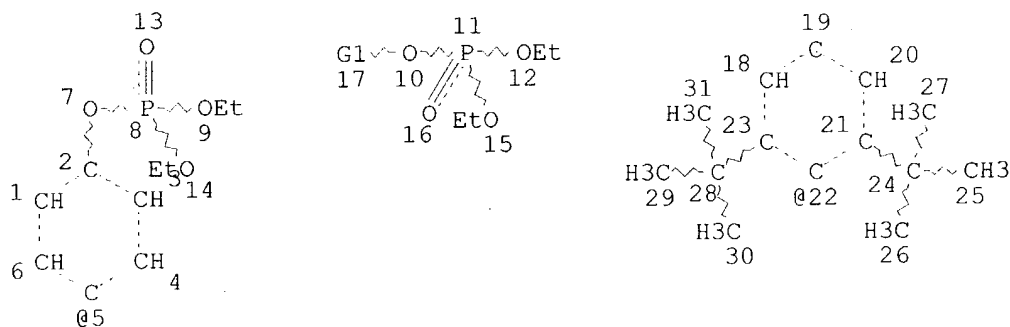
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 L3 STR



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 DEFAULT ECLEVEL IS LIMITED

GRAPH ATTRIBUTES:  
 RING(S) ARE ISOLATED OR EMBEDDED  
 NUMBER OF NODES IS 31

STEREO ATTRIBUTES: NONE  
 L5 14 SEA FILE=REGISTRY SSS FUL L3  
 L11 STR



VAR G1=5/22

NODE ATTRIBUTES:

DEFAULT MLEVEL IS ATOM

DEFAULT ECLEVEL IS LIMITED

GRAPH ATTRIBUTES:

RSPEC I

NUMBER OF NODES IS 31

STEREO ATTRIBUTES: NONE

L12 2 SEA FILE=REGISTRY SUB=L5 SSS FUL L11

L13 11 SEA FILE=HCAPLUS ABB=ON PLU=ON L12

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L13 ANSWER 1 OF 11 HCAPLUS COPYRIGHT 2004 ACS on STN

ACCESSION NUMBER: 2003:792234 HCAPLUS

DOCUMENT NUMBER: 140:42905

TITLE: Selective phosphorylation of hydroxyphenols for forming reactive flame retardants

AUTHOR(S): Toldy, A.; Anna, P.; Marosi, Gy.; Keglevich, Gy.; Almeras, X.; Le Bras, M.

CORPORATE SOURCE: Department of Organic Chemical Technology, Budapest University of Technology and Economics, Budapest, 1521, Hung.

SOURCE: Polymer Degradation and Stability (2003), 82(2), 317-323

CODEN: PDSTDW; ISSN: 0141-3910

PUBLISHER: Elsevier Science B.V.

DOCUMENT TYPE: Journal

LANGUAGE: English

AB Monophosphorylated hydroxy phenols were synthesized in a selective way and incorporated in different percentages into epoxy resins as reactive flame retardants. The LOI values of the epoxy resin samples could be increased this way up to 28%. The DSC results show that by increasing the percentage of incorporated monophosphorylated hydroquinone (HMP), the exothermic effect of the curing is decreased, which implies lower network d. An optimum balance is necessary between the flame retardant effect, detd. by percentage of HMP, and the lower degree of crosslinking.

IT 57246-14-7P

RL: SPN (Synthetic preparation); PREP (Preparation)

(diphosphorylated hydroquinone; selective phosphorylation of hydroxyphenols for forming reactive flame retardants)

REFERENCE COUNT: 11 THERE ARE 11 CITED REFERENCES AVAILABLE FOR THIS

## RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L13 ANSWER 2 OF 11 HCAPLUS COPYRIGHT 2004 ACS on STN  
 ACCESSION NUMBER: 2003:5302 HCAPLUS  
 DOCUMENT NUMBER: 138:58934  
 TITLE: Thermal runaway inhibitors for batteries  
 INVENTOR(S): Mandal, Braja K.; Filler, Robert  
 PATENT ASSIGNEE(S): USA  
 SOURCE: U.S. Pat. Appl. Publ., 19 pp.  
 CODEN: USXXCO  
 DOCUMENT TYPE: Patent  
 LANGUAGE: English  
 FAMILY ACC. NUM. COUNT: 1  
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 2003003358	A1	20030102	US 2001-879633	20010612
PRIORITY APPLN. INFO.:			US 2001-879633	20010612 ✓
OTHER SOURCE(S): MARPAT 138:58934				
AB The present invention provides for a battery having an anode, a cathode, and a flame-retarding electrolyte with a cond. greater than about 10-3. S/cm at ambient temp. and which includes a compd. that chem. interferes with flame propagation. The compd. that comprises the thermal runaway inhibitor has the formula $Z(P:XOR1OR2)_m$ where R1 and R2 are substituted alkyl, substituted aryl or trialkylsilyl; X is O or S; Z is selected from a substituted amino, amido or imido moiety or a silane; and m = 1-4.				
IT 57246-14-7P 479025-43-9P				
RL: MOA (Modifier or additive use); SPN (Synthetic preparation); PREP (Preparation); USES (Uses) (thermal runaway inhibitors for batteries)				

L13 ANSWER 3 OF 11 HCAPLUS COPYRIGHT 2004 ACS on STN  
 ACCESSION NUMBER: 2002:266488 HCAPLUS  
 DOCUMENT NUMBER: 137:79028  
 TITLE: A study on the selective phosphorylation and phosphinylation of hydroxyphenols  
 AUTHOR(S): Marosi, Gyorgy; Toldy, Andrea; Parlagh, Gyula; Nagy, Zoltan; Ludanyi, Krisztina; Anna, Peter; Keglevich, Gyorgy  
 CORPORATE SOURCE: Department of Organic Chemical Technology, Budapest University of Technology and Economics, Budapest, 1521, Hung.  
 SOURCE: Heteroatom Chemistry (2002), 13(2), 126-130  
 CODEN: HETCE8; ISSN: 1042-7163  
 PUBLISHER: John Wiley & Sons, Inc.  
 DOCUMENT TYPE: Journal  
 LANGUAGE: English  
 OTHER SOURCE(S): CASREACT 137:79028  
 AB By choice of appropriate reaction conditions, the phosphorylation of hydroquinone by di-Et chlorophosphate gave predominantly the monophosphate,  $HOC_6H_4OP(O)(OEt)_2$ -4. A similar reaction of phloroglucinol [1,3,5-C<sub>6</sub>H<sub>3</sub>(OH)<sub>3</sub>] led to the mixt. of the possible products (mono-, di- and triphosphates 6, 7, and 8). The monophosphinylation of the above hydroxyphenols by diphenylphosphinyl chloride could be accomplished with a good selectivity to give product 4 or 9, the yields, however, being variable.  
 IT 57246-14-7P  
 RL: SPN (Synthetic preparation); PREP (Preparation)  
 (prepn. of)  
 REFERENCE COUNT: 18 THERE ARE 18 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L13 ANSWER 4 OF 11 HCAPLUS COPYRIGHT 2004 ACS on STN  
 ACCESSION NUMBER: 2002:159173 HCAPLUS  
 DOCUMENT NUMBER: 136:340767  
 TITLE: Phenols as Starting Materials for the Synthesis of Arylstannanes via SRN1  
 AUTHOR(S): Chopa, Alicia B.; Lockhart, Maria T.; Dorn, Viviana B.  
 CORPORATE SOURCE: INIQO, Departamento de Quimica e Ingenieria Quimica, Universidad Nacional del Sur, Bahia Blanca, 8000, Argent.  
 SOURCE: Organometallics (2002), 21(7), 1425-1429  
 CODEN: ORGND7; ISSN: 0276-7333  
 PUBLISHER: American Chemical Society  
 DOCUMENT TYPE: Journal  
 LANGUAGE: English  
 OTHER SOURCE(S): CASREACT 136:340767

AB Phenols are converted into aryl di-Et phosphate esters (ArDEP), which on reaction with sodium trimethylstannide (1) or sodium triphenylstannide (2) in liq. ammonia afford arylstannanes by the SRN1 mechanism. Thus, the photostimulated reaction of phenylDEP (3), (4-methoxyphenyl)DEP (4), (4-biphenyl)DEP (5), (1-naphthyl)DEP (6), (2-naphthyl)DEP (7), and 2-(34), 3-(32), and (4-pyridyl)DEP (35) with 1 leads to monostannylated product in fair to excellent yields (20-98%). Also, substrates contg. two or three leaving groups react with 1 under irradiation, affording the corresponding di- or trisubstituted aryl compds. With tetra-Et m-phenylene bisphosphate (15), tetra-Et p-phenylene bisphosphate (21), (4-chlorophenyl)DEP (22), and 1,3,5-tris(diethylphospho)benzene (30), the di- or trisubstitution products 1,3-bis(trimethylstannyl)benzene (19) (79%), 1,4-bis(trimethylstannyl)benzene (23) (95 and 97%), and 1,3,5-tris(trimethylstannyl)benzene (31) (57%) are obtained, resp. Also, the reaction of 6 and 7 with 2 leads to substitution products in quant. yields, and the reaction of 21, 22, and (4-bromophenyl)DEP (24) with 2 affords 1,4-bis(triphenylstannyl)benzene (38) in high yields (70-100%). On the other hand, the results obtained in the photostimulated reaction of 24 and (4-iodophenyl)DEP (25) with 1, as well as in the reaction of 25 with 2, clearly indicate a fast HME reaction.

IT 57246-14-7  
 RL: RCT (Reactant); RACT (Reactant or reagent)  
 (photochem. stannylation of)

REFERENCE COUNT: 18 THERE ARE 18 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

2. L13 ANSWER 5 OF 11 HCAPLUS COPYRIGHT 2004 ACS on STN  
 ACCESSION NUMBER: 2002:85992 HCAPLUS  
 DOCUMENT NUMBER: 136:385894  
 TITLE: Approaches to the anodic generation of quinodimethanes  
 AUTHOR(S): Scholten, C.; Kuljanac, E.; Steckhan, E.; Utley, J. H. P.  
 CORPORATE SOURCE: Department of Chemistry, Queen Mary and Westfield College (University of London), London, E1 4NS, UK  
 SOURCE: Proceedings - Electrochemical Society (2001), 2001-14(Reactive Intermediates in Organic and Biological Electrochemistry), 73-76  
 CODEN: PESODO; ISSN: 0161-6374  
 PUBLISHER: Electrochemical Society  
 DOCUMENT TYPE: Journal  
 LANGUAGE: English  
 OTHER SOURCE(S): CASREACT 136:385894

AB Bis(carboxymethyl)arenes, i.e., anthracene-9,10-diylldiacetic acid and 1,4-dimethoxyphenylene-2,3-diacetic acid, and bis(trimethylsilylmethyl)arenes, i.e., 1,4-bis(trimethylsilylmethyl)benzene and 1,2-bis(trimethylsilylmethyl)benzene, were synthesized in moderate to good yields. In principle, bis(carboxymethyl)arenes are precursors for an anodic route to quinodimethanes via pseudo-Kolbe electrolysis.

Bis(trimethylsilylmethyl)arenes can, in principle, also be oxidatively cleaved to form quinodimethanes. We find that only products of reactions with nucleophilic solvents could be isolated, and we conclude that this potential anodic route to polymers or Diels-Alder adducts is impracticable.

IT 57246-14-7P

RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)

(approaches to anodic generation of quinodimethanes)

REFERENCE COUNT: 11 THERE ARE 11 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

✓ L13 ANSWER 6 OF 11 HCAPLUS COPYRIGHT 2004 ACS on STN

ACCESSION NUMBER: 2000:321770 HCAPLUS

DOCUMENT NUMBER: 133:43593

TITLE: Two-Step Synthesis of Arylstannanes from Phenols

AUTHOR(S): Chopa, Alicia B.; Lockhart, Maria T.; Silbestri, Gustavo

CORPORATE SOURCE: INIQC Departamento de Quimica e Ingenieria Quimica, Universidad Nacional del Sur, Bahia Blanca, 8000, Argent.

SOURCE: Organometallics (2000), 19(12), 2249-2250

CODEN: ORGND7; ISSN: 0276-7333

PUBLISHER: American Chemical Society

DOCUMENT TYPE: Journal

LANGUAGE: English

OTHER SOURCE(S): CASREACT 133:43593

AB Phenols are converted into aryl di-Et phosphates, which on reaction with alkali-metal triorganostannides in liq. NH<sub>3</sub> afford arylstannanes in excellent yield. Thus, ROP(O)(OEt)<sub>2</sub> was treated with R<sub>1</sub>SnNa in NH<sub>3</sub> to give 70-100% RSnR<sub>1</sub>3 (R = 1-, 2-naphthyl, 4-BrC<sub>6</sub>H<sub>5</sub>, R<sub>1</sub> = Ph; R = p-anisyl, 1-naphthyl, 4-ClC<sub>6</sub>H<sub>5</sub>, R<sub>1</sub> = Me). Similarly prepd. were 100% 1,4-(Ph<sub>3</sub>Sn)<sub>2</sub>C<sub>6</sub>H<sub>4</sub> and 95% 1,4-(Me<sub>3</sub>Sn)<sub>2</sub>C<sub>6</sub>H<sub>4</sub>.

IT 57246-14-7, Tetraethyl 1,4-phenylene diphosphate

RL: RCT (Reactant); RACT (Reactant or reagent)

(two-step synthesis of arylstannanes from phenols)

REFERENCE COUNT: 9 THERE ARE 9 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

✓ L13 ANSWER 7 OF 11 HCAPLUS COPYRIGHT 2004 ACS on STN

ACCESSION NUMBER: 1998:680082 HCAPLUS

DOCUMENT NUMBER: 129:303218

TITLE: Synthesis of condensed phosphates as flame retardant for polymers

AUTHOR(S): Wang, Xiaomei; Yang, Ping; Wang, Xikui; Zhu, Jiahou; Xiong, Wen

CORPORATE SOURCE: Department of Applied Chemistry, Shandong Institute of Building Materials, Jinan, 250022, Peop. Rep. China

SOURCE: Shandong Jiancai Xueyuan Xuebao (1998), 12(1), 25-27

CODEN: SJAXEU; ISSN: 1002-3046

PUBLISHER: Shandong Jiancai Xueyuan Xuebao Bianjibu

DOCUMENT TYPE: Journal

LANGUAGE: Chinese

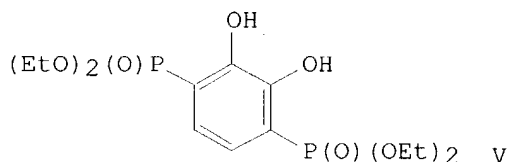
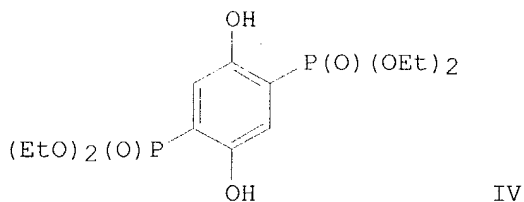
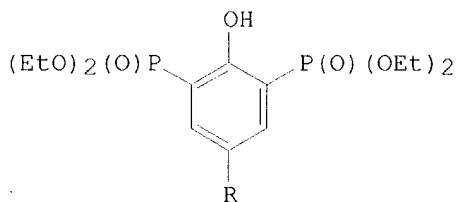
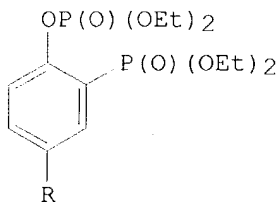
AB Twelve title compds. were synthesized using PCl<sub>3</sub>, in a C<sub>16</sub>H<sub>33</sub>NMe<sub>3</sub>Br catalytic solid-liq. phase transfer reaction in CCl<sub>4</sub>, and POCl<sub>3</sub> as starting materials, resp. The properties of the compds. as flame retardants for epoxy were studied. Exptl. results showed that the higher the content of P in the compds. the better the effect of flame retardance.

IT 57246-14-7P

RL: IMF (Industrial manufacture); TEM (Technical or engineered material use); PREP (Preparation); USES (Uses)

(synthesis of condensed phosphates as flame retardant for polymers)

L13 ANSWER 8 OF 11 HCAPLUS COPYRIGHT 2004 ACS on STN  
 ACCESSION NUMBER: 1984:571359 HCAPLUS  
 DOCUMENT NUMBER: 101:171359  
 TITLE: o-Hydroxyaryl diphosphonic acids  
 AUTHOR(S): Dhawan, Balram; Redmore, Derek  
 CORPORATE SOURCE: Petrolite Corp., St. Louis, MO, 63119, USA  
 SOURCE: Journal of Organic Chemistry (1984), 49(21), 4018-21  
 CODEN: JOCEAH; ISSN: 0022-3263  
 DOCUMENT TYPE: Journal  
 LANGUAGE: English  
 OTHER SOURCE(S): CASREACT 101:171359  
 GI



AB p-RC<sub>6</sub>H<sub>4</sub>OP(O)(OEt)<sub>2</sub> (R = H, OMe) rearranged with LiN(CHMe<sub>2</sub>)<sub>2</sub> to give 5,2-R(HO)C<sub>6</sub>H<sub>3</sub>P(O)(OEt)<sub>2</sub> (I; same R). Treating I with HOP(OEt)<sub>2</sub> in the presence of Et<sub>3</sub>N gave the phosphate II. Treating II with LiN(CHMe<sub>2</sub>)<sub>2</sub> gave the hydroxy-substituted bisphosphonates III. Similarly, IV and V were prep'd. from hydroquinone and pyrocatechol. Treating III-V with Me<sub>3</sub>SiCl and NaI in MeCN yielded trimethylsilyl phosphonate ester transesterification products that readily undergo hydrolysis to the corresponding phosphonic acids on contact with H<sub>2</sub>O at room temp.

IT **57246-14-7P**  
 RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)  
 (prepn. and rearrangement of, in presence of lithium diisopropylamide)

L13 ANSWER 9 OF 11 HCAPLUS COPYRIGHT 2004 ACS on STN  
 ACCESSION NUMBER: 1982:142410 HCAPLUS  
 DOCUMENT NUMBER: 96:142410  
 TITLE: Nickel-catalyzed cross-coupling of aryl phosphates with Grignard and organoaluminum reagents. Synthesis of alkyl-, alkenyl-, and arylbenzenes from phenols  
 AUTHOR(S): Hayashi, Tamio; Katsuro, Yoshio; Okamoto, Yasuo; Kumada, Makoto



CORPORATE SOURCE: Dep. Synth. Chem., Kyoto Univ., Kyoto, 606, Japan  
 SOURCE: Tetrahedron Letters (1981), 22(44), 4449-52  
 CODEN: TELEAY; ISSN: 0040-4039  
 DOCUMENT TYPE: Journal  
 LANGUAGE: English  
 OTHER SOURCE(S): CASREACT 96:142410  
 AB ROP(O)(OEt)<sub>2</sub> [I; R = 1- and 2-C<sub>10</sub>H<sub>7</sub> (= naphthyl), 4-R<sub>1</sub>C<sub>6</sub>H<sub>4</sub>; R<sub>1</sub> = H, Ph, (EtO)<sub>2</sub>P(O)O, MeO], prep'd. from ROH by sequential treatment with NaH/THF and ClP(O)(OEt)<sub>2</sub>, were converted into RR<sub>2</sub> (R<sub>2</sub> = alkyl, alkenyl, aryl) in high yields by cross-coupling with Grignard and organoaluminum reagents in the presence of Ni catalysts. E.g., I (R = 1-C<sub>10</sub>H<sub>7</sub>) was added to a mixt. of Ni(acac)<sub>2</sub> (acac = acetylacetonato) and Me<sub>3</sub>SiCH<sub>2</sub>MgCl in Et<sub>2</sub>O; the mixt. was stirred at room temp. for 10 h, then hydrolyzed with dil. HCl to give 79% 1-C<sub>10</sub>H<sub>7</sub>CH<sub>2</sub>SiMe<sub>3</sub>.  
 IT **57246-14-7P**  
 RL: SPN (Synthetic preparation); PREP (Preparation)  
 (prepn. and cross-coupling reaction of, with Grignard and organoaluminum reagents, nickel-catalyzed)

L13 ANSWER 10 OF 11 HCAPLUS COPYRIGHT 2004 ACS on STN  
 ACCESSION NUMBER: 1975:592819 HCAPLUS  
 DOCUMENT NUMBER: 83:192819  
 TITLE: Aromatic bisphosphates  
 INVENTOR(S): Kobayashi, Etsuro; Kamagami, Saburo  
 PATENT ASSIGNEE(S): Agency of Industrial Sciences and Technology, Japan  
 SOURCE: Jpn. Kokai Tokkyo Koho, 3 pp.  
 CODEN: JKXXAF  
 DOCUMENT TYPE: Patent  
 LANGUAGE: Japanese  
 FAMILY ACC. NUM. COUNT: 1  
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
JP 50093949	A2	19750726	JP 1973-143645	19731224

PRIORITY APPLN. INFO.: JP 1973-143645 19731224  
 GI For diagram(s), see printed CA Issue.  
 AB Arom. diols are heated with 2 moles POCl<sub>3</sub> to give bis(phosphoryl chlorides) Y(OP(O)Cl<sub>2</sub>)<sub>2</sub> (Y = arylene), which are treated with active H-contg. compds. HR (R = inorg. or org. residue), giving title bisphosphates Y[OP(O)R<sub>2</sub>]<sub>2</sub>. The products, e.g. I, are flame retardants. Thus, heating 34.2 g bisphenol A and 46 g POCl<sub>3</sub> at 140-330.degree. for 9 hr gave 67.9 g intermediate chloride, which (48 g) was refluxed with 75 ml MeOH for 4 hr to give I (R = OMe). Also prep'd. were p-C<sub>6</sub>H<sub>4</sub>[OP(O)(OEt)<sub>2</sub>]<sub>2</sub> and I (R = OEt, NH<sub>2</sub>).  
 IT **57246-14-7P**  
 RL: SPN (Synthetic preparation); PREP (Preparation)  
 (prepn. of)

L13 ANSWER 11 OF 11 HCAPLUS COPYRIGHT 2004 ACS on STN  
 ACCESSION NUMBER: 1957:43185 HCAPLUS  
 DOCUMENT NUMBER: 51:43185  
 ORIGINAL REFERENCE NO.: 51:8028a-g  
 TITLE: Preparation of chlorides and esters of aryl phosphates. Catalytic effects in the reaction of phenols with phosphorus oxychloride  
 AUTHOR(S): Katyrshkina, V. V.; Kraft, M. Ya.  
 CORPORATE SOURCE: S. Ordzhonikidze All-Union Chem. Pharm. Research Inst., Moscow  
 SOURCE: Zhurnal Obshchei Khimii (1956), 26, 3060-6  
 CODEN: ZOKHA4; ISSN: 0044-460X  
 DOCUMENT TYPE: Journal  
 LANGUAGE: Unavailable

OTHER SOURCE(S): CASREACT 51:43185

AB While many phenols react very slowly or not at all with a large excess of POC13 owing to the necessarily low reflux temp. of such mixts. the addn. of salts of the alkali metals catalyzes the reaction very greatly, the degree of catalysis increasing with the at. no. of the alkali metal as well as with the acidic dissocn. const. of the specific phenol. The explanation of the catalysis is the possible equil. between such salts as NaCl and ArOH, in which ArONa and HCl are formed; owing to insoly. of HCl in refluxing POC13, the equil. would tend to shift toward formation of RONA which would instantly react with POC13. The catalytic effect in this reaction with a variety of phenols is shown graphically with salts such as BaCl2, NaCl, KCl, RbCl, CsCl, and FeCl3, which are arranged in ascending order of catalytic activity; ZnCl2 and CoCl2 also show such activity. Refluxing 94 g. PhOH and 96 ml. POC13 10 hrs. gave 35.9 g. HCl, 168.3 g. PhOPOC12, and 21.3 g. crude (PhO)2POCl. Refluxing 94 g. PhOH, 549 ml. POC13, and 5 g. KCl 10 hrs. gave 95.8% PhOPOC12, b7 106-7.5.degree.. While p-O2NC6H4OH failed to react on refluxing with 6 moles POC13, addn. of 0.5 g. NaCl in 3.5 hrs. gave 99.9% evolution of HCl and distn. gave 88% p-O2NC6H4OPOC12, b2 154-5.degree.. This (33.6 g.) was treated with cooling with 30 ml. dry EtOH, kept overnight, and distd. in vacuo after washing with H2O and NaOAc and extn. with (CH2Cl)2, yielding 77% p-O2NC6H4OPO(OEt)2 (Fosfakol), b1 170-1.degree., d20 1.2782, nD20 1.5080; the yield was 70% if undistd. dichloride is used. The following ROPOCl2 are prepd. similarly [R (catalyst), yield, b.p., (yield, b.p., d20, nD20 of di-Et ester) given]: o-MeC6H4 (NaCl), 91%, b8 118.degree. (52%, b1.5 119.degree., 1.1313, 1.4812); p-iso-PrC6H4 (NaCl), 88.5%, b11 145-6.degree. (83%, b10 174.5-5.5.degree., 1.0852, 1.4770); p-Me2EtCC6H4 (KCl), 89.3%, b11 155-9.degree. (76.1%, b0.5 144-6.degree., 1.0640, 1.4838); p-C7H15C6H4 (KCl), 84.8%, b1.5 167-9.degree. (71.8%, b2 178-9.degree., 1.0328, 1.4761); p-Me2BuCC6H4 (KCl), 86.5%, b0.5 123-5.degree. (79.8%, b1 164-7.degree., 1.1380, 1.4812); p-C9H19C6H4 (KCl), 89%, b0.5 167.degree. (77.6%, b1 181.5-4.degree., 1.0125, 1.4765); p-C12H25C6H4 (KCl), 80.7%, b1 204-8.degree. (78%, b0.5 204-7.degree., 0.9673, 1.4750); o-MeOC6H4 (FeCl3), 89%, b2 120.degree. (75.3%, b1.5 144.degree., 1.1872, 1.4972); m-MeOC6H4 (KCl), 89.2%, b1.5 123-4.degree. (76%, b1.5 154-6.degree., 1.1750, 1.4874); p-MeOC6H4 (NaCl), 88.3%, b1.5 118-20.degree. (55%, b1 148.degree. 1.1757, 1.4865); o-O2NC6H4 (KCl), 83%, b1 138-9.degree. (76%, b1 158-60.degree. 1.2761, 1.4979); m-O2NC6H4 (NaCl), 87%, b1 144.degree. (72%, b0.5 155-8.degree. 1.2745, 1.4991); p-O2NC6H4 (NaCl), 88.1%, b1 144.degree. (77%, b1 170-1.degree., 1.2782, 1.5080); 2,4-Me(O2N)C6H3 (NaCl), 90.1%, - (73.4%, b0.5 164-6.degree. 1.2516, 1.5102); 2,4-MeO(O2N)C6H3 (NaCl), 88.2%, b2 169-71.degree. (65%, b1 187.degree. 1.2950, 1.5141); 1-C10H7 (KCl), 85%, b0.5 138-40.degree. (71.9%, b0.5 151-4.degree., -, 1.5245); 2-C10H7 (KCl), 84.6%, b1 155-6.degree. (72.0%, b1 170.degree., 1.1792, 1.5250); p-C6H4 (KCl), 85.6%, b20 212.degree. (73%, b1 184.degree., 1.2789, 1.4725); m-C6H4 (KCl), 53%, b1 157.degree.. The last 2 substances are bis-phosphoryl derivs. The p-alkylphenyl derivs. listed in the table above contain straight chain alkyl groups, being prepd. from the appropriate p-acylphenols by Clemmensen reduction.

IT 57246-14-7, Ethyl p-phenylene phosphate  
(prepn. of)

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=> fil caold

FILE 'CAOLD' ENTERED AT 12:29:28 ON 28 JAN 2004

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FILE LAST UPDATED: 01 May 1997 (19970501/UP)

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=> s l12
L14      1 L12

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=> d all l14 1

L14 ANSWER 1 OF 1 CAOLD COPYRIGHT 2004 ACS on STN
AN CA51:8028a CAOLD
TI prepn. of chlorides and esters of aryl phosphates-catalytic effects in the
reaction of phenols with P oxychloride
AU Katyshkina, V. V.; Kraft, M. Ya.
IT 311-45-5 597-87-5 770-12-7 1089-14-1 6964-36-9 13538-32-4
13929-83-4 16462-76-3 16519-26-9 20464-67-9 20464-68-0 33650-14-5
33965-78-5 38135-34-1 38815-40-6 55231-78-2 57246-14-7
63228-12-6 67951-86-4 99981-34-7 101259-60-3 101571-78-2 101571-79-3
102944-78-5 109507-14-4 109507-15-5 109593-83-1 115308-11-7 117043-13-7
117043-15-9
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Property values tagged with IC are from the ZIC/VINITI data file provided by InfoChem.

STRUCTURE FILE UPDATES: 27 JAN 2004 HIGHEST RN 642407-31-6  
DICTIONARY FILE UPDATES: 27 JAN 2004 HIGHEST RN 642407-31-6

TSCA INFORMATION NOW CURRENT THROUGH JULY 14, 2003

Please note that search-term pricing does apply when conducting SmartSELECT searches.

Crossover limits have been increased. See HELP CROSSOVER for details.

Experimental and calculated property data are now available. For more information enter HELP PROP at an arrow prompt in the file or refer to the file summary sheet on the web at:

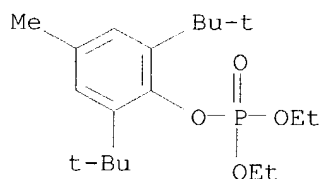
<http://www.cas.org/ONLINE/DBSS/registryss.html>

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=> d ide can l12 tot

L12 ANSWER 1 OF 2 REGISTRY COPYRIGHT 2004 ACS on STN  
 RN 479025-43-9 REGISTRY  
 CN Phosphoric acid, 2,6-bis(1,1-dimethylethyl)-4-methylphenyl diethyl ester  
 (9CI) (CA INDEX NAME)  
 FS 3D CONCORD  
 MF C19 H33 O4 P  
 SR CA  
 LC STN Files: CA, CAPLUS, USPATFULL

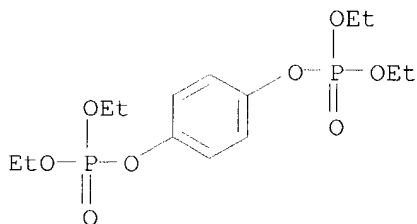


\*\*PROPERTY DATA AVAILABLE IN THE 'PROP' FORMAT\*\*

1 REFERENCES IN FILE CA (1907 TO DATE)  
 1 REFERENCES IN FILE CAPLUS (1907 TO DATE)

REFERENCE 1: 138:58934

L12 ANSWER 2 OF 2 REGISTRY COPYRIGHT 2004 ACS on STN  
 RN 57246-14-7 REGISTRY  
 CN Phosphoric acid, 1,4-phenylene tetraethyl ester (9CI) (CA INDEX NAME)  
 OTHER CA INDEX NAMES:  
 CN Ethyl p-phenylene phosphate (6CI)  
 OTHER NAMES:  
 CN Tetraethyl 1,4-phenylene diphosphate  
 FS 3D CONCORD  
 MF C14 H24 O8 P2  
 LC STN Files: BEILSTEIN\*, CA, CAOLD, CAPLUS, CASREACT, USPATFULL  
 (\*File contains numerically searchable property data)



\*\*PROPERTY DATA AVAILABLE IN THE 'PROP' FORMAT\*\*

11 REFERENCES IN FILE CA (1907 TO DATE)  
 11 REFERENCES IN FILE CAPLUS (1907 TO DATE)  
 1 REFERENCES IN FILE CAOLD (PRIOR TO 1967)

REFERENCE 1: 140:42905  
REFERENCE 2: 138:58934  
REFERENCE 3: 137:79028  
REFERENCE 4: 136:385894  
REFERENCE 5: 136:340767  
REFERENCE 6: 133:43593  
REFERENCE 7: 129:303218  
REFERENCE 8: 101:171359  
REFERENCE 9: 96:142410  
REFERENCE 10: 83:192819